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Docket No.: G0365.0379  
(PATENT)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of:  
Soren L. Jensen

Application No.: 10/560,061

Confirmation No.: 5308

Filed: December 13, 2005

Art Unit: Not Yet Assigned

For: MINERAL FIBRE PRODUCTS

Examiner: Not Yet Assigned

**CLAIM FOR PRIORITY AND SUBMISSION OF DOCUMENTS**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

Applicant hereby claims priority under 35 U.S.C. 119 based on the following prior foreign applications filed in the following foreign countries on the dates indicated:

<u>Country</u>	<u>Application No.</u>	<u>Date</u>
United Kingdom	0313763.5	June 13, 2003
United Kingdom	0317989.2	July 31, 2003



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In support of this claim, a certified copy of each said original foreign application is filed herewith.

Dated: August 15, 2006

Respectfully submitted,

By   
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For Innovation

The Patent Office  
Concept House  
Cardiff Road  
Newport  
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NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Consumer Empowerment Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

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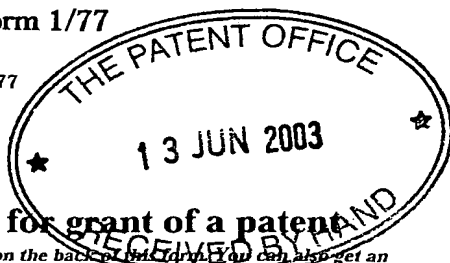
Signed

*William Morell*

Dated 10 May 2006

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# Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

The Patent Office

Cardiff Road  
Newport  
South Wales  
NP9 1RH

1. Your reference

PRL04521GB

13 JUN 2003

2. Patent application number

(The Patent Office will fill in this part)

0313763.5

3. Full name, address and postcode of the or of each applicant (*underline all surnames*)

ROCKWOOL INTERNATIONAL A/S  
584 Hovedgaden  
DK-2640 Hedehusene  
Denmark

Patents ADP number (*if you know it*)

If the applicant is a corporate body, give the country/state of its incorporation

DK

691188006

4. Title of the invention

Mineral Fibres

5. Name of your agent (*if you have one*)

Gill Jennings & Every

"Address for service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)

Broadgate House  
7 Eldon Street  
London  
EC2M 7LH

Patents ADP number (*if you know it*)

745002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (*if you know it*) the or each application number

Country

Priority application number  
(*if you know it*)

Date of filing  
(*day / month / year*)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(*day / month / year*)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (*Answer 'Yes' if:*

YES

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

**Patents Form 1/77**

9. Enter the number of sheets for any of the following items you are filing with this form.  
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Continuation sheets of this form

Description	6
Claim(s)	1
Abstract	1
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Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*)

Request for substantive examination (*Patents Form 10/77*)

Any other documents  
(please specify)

NO

11. For the applicant  
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature

Date

13 June 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

LAWRENCE, Peter Robin Broughton

020 7377 1377

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Rockwool International A/S

PRL04521GB

**Mineral Fibres**

It has been known for a long time that some bonded mineral wool products do not give as good fire protection as would theoretically be expected. An example is fire doors, which are semi-sealed products containing bonded mineral wool. Other products in which a mineral insulation is wholly or partially enclosed, e.g., within sheets of material which prevent free access of air, give similar problems. It is also known that if a thick slab of bonded, high density, mineral wool is exposed to very high temperature, the core tends to collapse due to sintering, to leave a void, and similar collapse may be the cause of the problem with fire doors. This is especially a problem when the binder is organic.

Typical stone or rock mineral fibres with which we are concerned in this invention contain at least 1%, and preferably at least 2, 3, 4 or 5% or more ferrous (as oxide). The fibres often contain ferric to a total iron content of at least 3% and usually 5 to 10%. They also contain usually at least 8%, often 10-20%, or more, CaO and at least 5%, often 8 to 15% or more MgO. The fibres generally contain  $\text{Na}_2\text{O} + \text{K}_2\text{O}$  in amounts of from zero to 8%, usually 1-4% or 5%. The amount of  $\text{SiO}_2$  is usually 35-55%, often 38-50%. The amount of  $\text{Al}_2\text{O}_3$  is usually 10 to 25%, often 13% to 22%. All analyses are expressed by weight of oxides.

It has previously been thought that these problems were due to the exotherm of the combustion of the bonding agent creating so much local heating as to cause severe sintering.

We have now discovered that the problem does not arise from the simple temperature effect caused by the exotherm but instead arises because of the reducing conditions created by the combustion of the binder under conditions where there is inadequate oxygen.

We have established that if rock fibres are heated above  $T_g$  in the presence of ample air then there is a tendency for the predominant effect to be surface crystallisation on the fibres of magnesium calcium silicates accompanied by embrittlement of the fibres, whilst if the heating is conducted under conditions without ample air the predominant effect is surface diffusion and sintering and collapse of the fibres.

The mechanism which distinguishes between this surface diffusion and sintering collapse on the one hand and surface crystallisation on the other is associated with the equilibrium between ferrous and ferric within the fibre chemical structure.

The problems discussed above of collapse of some bonded structures at high temperatures are avoided if surface crystallisation is maximised and surface diffusion and sintering minimised, and that this is also associated with the ferrous-ferric equilibrium.

This explains why iron has a uniquely high influence on the sintering properties of the fibres, relative to other elements conventionally in the fibres, since none of these other elements can participate easily in a similar equilibrium between different oxidation states.

It is well known that a substantial amount of the iron in the rock and other mineral fibres is usually ferrous, this amount perhaps being 70 or 80% when the fibres are made in a cupola or under other reducing conditions, and significantly less when the fibres are made under oxidising conditions.

The calcium and magnesium is thought to be held within the network by relatively weak bonding to oxygen atoms of the network. Under low and moderate temperatures the calcium and magnesium are held within the network but when the temperature becomes sufficiently high (for instance around  $550^{\circ}\text{C}$  for a fibre having a typical  $T_g$  of around  $670^{\circ}\text{C}$ ) the calcium or magnesium bonds with oxygen can tend to break in favour of the oxygen oxidising ferrous to

ferric, and with the calcium and magnesium cations diffusing through the solid network towards the surface. This occurs mainly when the surface is oxidising, and so the calcium and magnesium ions are oxidised. This appears  
5 to involve oxidation to calcium magnesium oxides but there may be some interaction with the silica.

When the fibre is subsequently heated to above  $T_g$ , the calcium and magnesium at the surface interact to form calcium magnesium silicate (diopside and augite) crystals.  
10 The surface enrichment on initial heat treatment thus increases the tendency to surface crystallisation when the fibres are subsequently heated to above  $T_g$ .

Some alkali metal will also sometimes leave its oxygen bonds behind also and also migrate to the surface in  
15 similar manner. Aluminium and titanium diffuse much less, and oxygen diffuses hardly at all below  $T_g$ .

This mechanism is consistent with the observations about inferior properties in fire doors and void formation in dense bonded products, when the binder burns off with an  
20 exotherm. This causes reducing conditions to prevail.

The mechanism is also consistent with observations as to the effect when mineral fibre are subjected to  $1,000^\circ\text{C}$  at atmospheric pressure in various atmospheres. When the atmosphere is totally air, there is no surface diffusion  
25 and sintering, but only surface crystallisation. When the atmosphere is nitrogen and a small amount ( $10^{-4}$ ) oxygen, the fibre collapses and sinters. When the atmosphere is hydrogen with  $10^{-18}$  oxygen, there is even greater collapse.

Accordingly, our understanding of the mechanisms  
30 discussed above makes it possible to minimise the risk of sintering causing collapse. In particular, we achieve this by subjecting the fibres to a preparatory treatment which is done for the purpose of promoting the diffusion of calcium and magnesium to the surface to increase their  
35 tendency to surface crystallisation. It is necessary to have a useful amount of ferrous iron in the fibres to be oxidised, to ferric, and to have an oxidising atmosphere

during the pretreatment around the fibre to oxidise the calcium and magnesium, to allow migration of the calcium and magnesium to the surface and subsequent oxidation at the surface.

5       Accordingly, we now provide fibres in which the calcium and magnesium has diffused, or will diffuse preferentially, to the surface of the fibres and crystallise there, together with oxidation of ferrous within the network to ferric.

10       The treated fibres have a higher concentration of calcium and magnesium at the surface with depletion within the fibre, and with the iron being predominantly (e.g., at least 70%, and usually at least 80, 90 or 95% and preferably substantially 100%) ferric within the fibre.

15       Fibres manufactured, for subsequent use, with these properties are novel materials.

      The invention includes the deliberate use of these fibres having surface enrichment of calcium and magnesium with a core in which the iron is mainly ferric as a  
20       starting material for the manufacture of insulation. For instance fibres having this structure may be converted into elements such as fire doors and sandwich panels.

      The enrichment generally involves the formation of a surface layer in which the concentration of CaO and of MgO  
25       is at last 1.5 times, and usually 2 to 4 or 5 times, the concentration in the total fibre. The thickness of the surface layer having this high enrichment is usually less than  $1\mu\text{m}$  or  $2\mu\text{m}$ , e.g., 0.02 to  $0.5\mu\text{m}$ .

      Generally there is also high surface enrichment of  
30       iron in the surface layer. This is usually depletion of alkali,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in the surface layer.

      The invention also relates to the prevention of the reducing conditions that prevail when binder is burnt off, for instance by ensuring that oxidising conditions prevail  
35       at all relevant temperatures.

      Generally the elements which are to be exposed to high temperatures should be provided with structural integrity

by using a non-combustible system for holding the fibres in place, for instance a silicate binder or some arrangement whereby the fibres are held in fixed positions without the need for a combustible binder. A coating may be applied to the fibres.

A preferred aspect of the invention is one in which the fibres are subjected to heating under oxidising conditions at a temperature which is sufficiently high to cause the diffusion of calcium and magnesium to the surface and the oxidation of ferrous to ferric but which preferably is insufficiently high to allow any significant diffusion at the surface. This can be done on the final shaped fibre batt or on free fibres. By this means the desired novel fibres having the surface concentration of calcium and magnesium and the core containing ferric will be provided without any risk of melt flow having occurred.

For instance for a typical stone wool with  $T_g$  around  $670^{\circ}\text{C}$  the diffusion of calcium and magnesium can start at around  $540^{\circ}\text{C}$  or  $550^{\circ}\text{C}$ , and crystallisation of the entire fire network may start at around  $750^{\circ}\text{C}$  with most crystallisation having occurred by about  $850^{\circ}\text{C}$ , and with  $T_g$  around  $670^{\circ}\text{C}$ . Accordingly heating these fibres in an oxidising atmosphere at temperatures of, for instance, 500 to 600 or  $650^{\circ}\text{C}$  or even up to around 700 or  $720^{\circ}\text{C}$  will achieve the surface enrichment in calcium and magnesium of the fibres without damaging the fibrous texture of the product and without allowing diffusion at the surface to occur.

In general, any temperature can be used provided it is above the temperature at which diffusion of calcium and magnesium and oxidation of ferrous becomes significant but below the temperature at which the fibres become unacceptably brittle due to crystallisation throughout the structure, and if the temperature is above  $T_g$  then the conditions must be such as to insure that surface diffusion does not occur during the heating. In practice the temperature for the pretreatment can be any temperature

between the temperature at which oxidation starts, up to the crystallisation temperature.

5 The pretreatment may be applied while the fibres are loose or while they are in a batt, provided the conditions are sufficiently oxidising. The final insulation articles may then be assembled from the treated fibres by conventional techniques.

10 The rate of increase of temperature during the treatment is preferably selected so as to optimise the amount of oxidation to ferric. If inadequate oxidation is occurring better results may be obtained by reducing the rate of increase. The duration of the treatment may be only a few seconds but is preferably at least 1 minute and usually at least 5 or 10 minutes. Typically it is 15  
15 minutes to 1½ or 2 hours or more.

The invention includes each of the various aspects discussed above, and also those summarised by the following claims.

CLAIMS

1. Mineral fibres suitable for subsequent incorporation into articles for use as insulation (such as fire doors and other products in which the fibres are wholly or partially enclosed) and which have a greater concentration of MgO and CaO on their surface than in their core and wherein iron is predominantly present as  $\text{Fe}_2\text{O}_3$ .  
5
2. A method of treating mineral fibres containing FeO, CaO and MgO to improve their properties, wherein the method comprises exposing the fibres under oxidising conditions to a temperature which is at or above a value at which oxidation of FeO to  $\text{Fe}_2\text{O}_3$  occurs (and preferably above  $T_g$ ) but which is below the onset of crystallisation.  
10
3. A method according to claim 2 in which the temperature is at least  $550^\circ\text{C}$ .  
15
4. Articles for use as insulation and made by wholly or partially enclosing fibres as defined in claim 1 or made by the method of claims 2 or 3.

## A B S T R A C T

Mineral Fibres

- 5        The properties of mineral fibreas ~~are~~ improved by exposure to a treatment which causes migration of CaO and MgO.
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